

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants:	Alexander P. Moravsky et al.	Examiner:	Stuart L. Hendrickson
Serial No.:	09/680,291	Art Unit:	1793
Filed:	October 6, 2000	Docket:	21088/14311
For:	DOUBLE-WALLED CARBON NANOTUBES AND METHODS FOR PRODUCTION AND APPLICATION		
Confirmation No.:	9193		

Commissioner for Patents
P. O. Box 1450
Alexandria, VA 22313-1450

DECLARATION OF ALEXANDER P. MORAVSKY

Sir:

I, Alexander P. Moravsky, hereby declare and state as follows:

1. I am one of the applicants of the above-identified application, and I have complete knowledge of all aspects of the application.
2. I am currently employed as Senior Scientist at MER Corporation, Tucson, Arizona, and have been so employed since 1999. Previous to that year, I assisted MER Corporation in Tucson, Arizona for several months in 1998 as a consultant. My employment history since 1975 is set forth in the Resume of Alexander P. Moravsky, attached hereto as Exhibit 1 and incorporated herein by reference and made a part hereof.
3. I am currently involved in fullerene and carbon nanotube materials synthesis and property studies, and have been so involved since 1991. I have extensive experience and many publications in this field, as set forth in my attached Resume in Exhibit 1.

4. I have earned Ph.D, M.Sc. and B.Sc. degrees in the fields of Chemistry, Physics and Molecular and Chemical Physics, all as set forth in my attached Resume in Exhibit 1.

5. The present application is directed to, *inter alia*, a solid substance comprised by more than one half by weight of hollow carbon nanotubes having walls consisting essentially of two layers of carbon atoms, said nanotubes consisting of two concentric nearly cylindrical graphene layers. It is also directed to, *inter alia*, to substantially pure double wall nanotubes, wherein the double-wall nanotubes have two concentric nearly cylindrical graphene layer.

6. I have been advised by counsel that the United States Patent and Trademark Office ("USPTO") has rejected Claims under 35 U.S.C. 103(a) as being unpatentable over an article by Flahaut et al. (Flahaut et al., J. Mater. Chem., 2000, 10, 249-252), further "Flahaut et al".

7. I have been advised by counsel that the Flahaut et al. article was published on January 27, 2000.

8. The present application was filed in the USPTO on October 6, 2000.

9. The invention described and claimed in the above-identified application was completed in the U.S. prior to January 27, 2000.

10. As evidence hereof, annexed hereto and made a part hereof are Exhibits 1-10. All of the acts described herein took place in either the United States or a NAFTA country other than the United States or a WTO member country prior to January 27, 2000, and were performed by myself or by scientists and/or technicians working under my direct supervision or control, except to the extent indicated herein. Data or information not pertinent to the invention and dates have been masked out in the preparation of their photocopies.

11. Attached hereto as Exhibit 2 is a copy of a notebook page, which outlines a synthesis for a product of the present invention. This outlines an arc discharge evaporation of a metal-graphite electrode, where the anode was a graphite rod (8.2 mm in diameter), having a

drilled channel of about 189 mm in length and 3.16 mm in diameter. The cathode was a graphite rod, which was positioned coaxially with the anode. The catalyst was prepared by heating nickel, cobalt and iron together with elemental sulfur. The resulting conglomerate was ground in a ball mill to a micron particle size and then intimately mixed with carbon powder. The channel drilled in the graphite rod was tightly filled with the mixture to obtain an anode containing 79.91% C, 10.26% Ni, 2.77% Co, 5.44% Fe and 1.62%S.

An arc discharge was carried out with an arc current of 81A at 350 Torr in an atmosphere of Argon (175 Torr) and hydrogen (175 Torr) (1:1). The voltage drop across the gap was 26-28 V, and the rod feed was 2 mm/min. The duration of the arc process was about 45 min.

The carbon material produced in the arc discharge was deposited on the walls of the reactor and on the electrodes wherefrom it was collected separately. A fibrous material (elastic web-like product) was collected from the cathode.

12. A SEM examination of the web-like products obtained from the process described in Paragraph 11 showed dominating amounts of curled fibers being present in the product.

13. Exhibit 3 is an example of a TEM image of the product obtained from the process described in Paragraph 11. As shown in Exhibit 3, unusually thick nanotubes of approximately 4 nm in diameter were observed, assembled in thin bundles containing a few tubes.

14. The image was quite different than the appearance of single walled nanotubes, commonly produced in arc discharge processes, whereby tubes are about 1.5 nm in diameter and the bundles contain some dozen tubes.

15. Further, the walls of the tubes in Fig. 3 are thicker than is observed for commonly produced SWNTs, which is consistent with the nanotubes having two walls.

16. TEM images made by an instrument with better resolution unambiguously show that the tubes produced by the process described hereinabove in paragraph 11 have a two-wall structure. The images are attached hereto as Exhibits 4-8. These images unambiguously show that the nanotubes have two concentric nearly cylindrical graphene layers.

17. Additional samples were prepared in accordance with the procedure outlined in Example 1.

18. Samples, which were prepared by me or under my direct supervision, in accordance with the procedure described in the underlying application, such as Example 1, were examined under HRTEM (High Resolution TEM) in Russia by my colleague, Dr. Krinichnaja. Attached as Exhibit 9 is an email message from Dr. Krinichnaja in Russian; Exhibit 10 is a translation thereof, which verifies that the sample which I provided to her and which she examined using TEM, consisted of double wall nanotubes, having diameters of 3.5 to 5.2 nm. Good quality HRTEM analysis of the samples produced prior to January 27, 2000 revealed that samples contained not only DWNTs as the dominant carbon product, and also that no SWNTs were detected.

19. As shown by all the data described herein, all of the experiments described herein produced a solid product comprised by more than one half by weight of hollow carbon nanotubes having walls consisting essentially of two layers of carbon atoms, consisting of two concentric nearly cylindrical graphene layers. The product produced was isolated and very pure double wall nanotubes, where the double wall nanotube have two nearly cylindrical graphene layers.

20. Thus, the acts described herein were completed prior to the January 27, 2000, the publication date of the Flahaut et al. article.

21. I further declare that all statements made herein of my knowledge are true and that all statements made on information and belief are believed to be true and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment or both under Section 1001 of Title 18 of the United States

Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: October 04, 2010

Alexander P. Moravsky
Alexander P. Moravsky

EXHIBIT

1

RESUME
ALEXANDER P. MORAVSKY

EDUCATION

Ph.D. Chemistry, Moscow Institute for Physics and Technology (MIPT), 1975

M.Sc. Physics of Fast Processes, MIPT, 1972

B.Sc. Molecular and Chemical Physics, MIPT, 1970

PROFESSIONAL EXPERIENCE

1998-Present MER Corporation, Senior Scientist, Tucson, AZ

1980-1998 Senior Research Fellow, Head of a Research Group, Institute of Problems of Chemical Physics, Chernogolovka, Russia

1979-1980 Post Doctorate of Prof. J.K.Stille, Colorado State Univ., Fort Collins, CO

1975-1979 Research Fellow, Institute of Chemical Physics, Chernogolovka, Russia

RECORD OF TECHNICAL ACHIEVEMENTS

- 11 Patents
- 240 papers in refereed journals
- 5 Reviews

INDUSTRIALLY IMPLEMENTED TECHNOLOGIES:

- technique for ketones and aldehydes micro-analysis
- methane absorption accumulators

AREAS OF TECHNICAL EXPERTISE

- R&D on advanced new materials, basic chemical technology, organic synthesis
- Catalysts, small molecule catalytic conversions of industrial interest
- Redox processes and fast reactions in solution and films
- Purification of substances, analytical chemistry
- Analysis of sophisticated chemical kinetic schemes
- Spectral analysis, NMR and ESR, XPS, GC, GC/MS, LC, HPLC
- Fullerene and nanocarbon materials science and technology

Dr. Moravsky A.P.
List of recent publications. 1993-2010.

1. "The Loss-Function of Solid C_{60} ", *Synthetic Metals*, v.56, No 2-3, 2961-2966 (1993). V.I.Rubtsov, Yu.M.Shulga, A.S.Lobach, and A.P.Moravskii.
- "Photophysical Properties of C_{60} . Picosecond Study of Intersystem Crossing", *J.Photochem.Photobiol. A: Chem.*, v.70, No2, 153-156 (1993). V.A.Nadtochenko, I.V.Vasil'ev, N.N.Denisov, I.V.Rubtsov, A.S.Lobach, A.P.Moravskii, and A.F.Shestakov.
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- "Photochemical Properties of C_{60} . Triplet Excited C_{60} Quenching by Electron Acceptors TCNQ and TCNE in Solution. Laser Photolysis Study", *Chem.Phys.Letters*, v.208, No5-6, 431-435 (1993). V.A.Nadtochenko, N.N.Denisov, I.V.Rubtsov, A.S.Lobach, and A.P.Moravskii.
- "Triplet Excited C_{60} Quenching by TCNE in Benzonitrile Solution. Formation of the Ion-Radical Pair [$C_{60}^{+...}TCNE$]", *Russian Chemical Bulletin*, v.42, N7, 1171-1173 (1993). V.A.Nadtochenko, N.N.Denisov, I.V.Rubtsov, A.S.Lobach, and A.P.Moravskii.
- "Photophysical Properties of Fullerenes. Picosecond and Nanosecond Transient Absorption Spectra of C_{60} and C_{70} . New Bands in the Near IR." *Fullerenes. The First Int. Interdisciplinary Colloquium on the Sci. and Tech. of Fullerenes*, 1993, Santa Barbara, USA, Abstract, p.116. Nadtochenko V.A. et al.
- "Photochemical Properties of C_{60} . Photooxidation of C_{60} by TCNE and TCNQ in Solutions. Laser Photolysis Study". *Fullerenes. The First Int. Interdisciplinary Colloquium on the Sci. and Tech. of Fullerenes*, 1993, Santa Barbara, USA, Abstract, p.192-194. Nadtochenko V.A. et al.
- "Photophysical and Photochemical Properties of C_{60} . Picosecond and Nanosecond Laser Photolysis Study", *International Workshop on Fullerenes and Atomic Clusters*, 1993, St.Peterburg, Russia, p.29. Nadtochenko V.A. et al.
- "On the Reaction of Buckminsterfullerene with Tetrabutylammonium Tetrahydroborate", *Russian Chemical Bulletin*, N3, 803(1993). V.D.Makhaev, Yu.M.Shulga, A.S.Lobach, V.N.Vasilets, O.S.Roshchupkina, and A.P.Moravskii.
10. "Reaction of Organic Cation Tetrahydroboronates with Fullerene C_{60} ", *Int. Conference "Physics and Chemistry of Fullerenes"*, Greece, 1993, Abstracts, p.34. V.D.Makhaev, A.S.Lobach, Yu.M.Shulga, O.S.Roshchupkina, and A.P.Moravskii.
- "Interaction of Methane with Acetylene in the Presence of Ziegler-Natta Type Catalysts", *Neftekhimiya*, v.32, N4, 324-330 (1992). E.M.Efimova, A.P.Moravskii, and N.F.Noskova.
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- "Reactions of Hydrocarbons with Electrophilic Transition Metal Complexes in Trifluoroacetic Acid Media", *Uspekhi Khimii*, v.63, N2, 130-144 (1994). N.F.Goldshleger, and A.P.Moravskii.
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- "Quenching of Triplet-Excited Fullerene C_{60} by TCQM in Solutions", *Zh.Fiz.Khimii*, v.68, N2, 228-231 (1994). V.A.Nadtochenko, N.N.Denisov, A.S.Lobach, and A.P.Moravskii.
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"Preparation and Single Crystal Structure Determination of the Intercalate C_{60} /TMPD", IW Fullerenes and Atomic Clusters, 1995, p.53-54, V.A.Nadtochenko, A.P.Moravsky, V.V.Gritsenko, G.V.Shilov, and O.A.Dyachenko.

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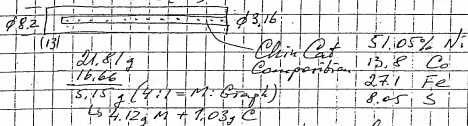
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EXHIBIT

2

Run CF39

H₂ 175 Torr (200 scan) + Ar 125 Torr (200 scan) = 350 TorrRod: 2202(800) I8tA ϕ 2.0 mm/wireSteady-state vaporization found at U_{26-28V} at $I_{8t} \approx 52.0$

Composition of the rod:

$$\begin{aligned} \text{Ni} &= 21.81g \Rightarrow \frac{21.81}{16.66} = 1.31 \Rightarrow 1.31 \times 1.03 = 1.35g \\ \text{Co} &= 16.66g \Rightarrow \frac{16.66}{16.66} = 1.00 \Rightarrow 1.00 \times 1.03 = 1.03g \\ \text{Fe} &= 5.15g \Rightarrow \frac{5.15}{16.66} = 0.31 \Rightarrow 0.31 \times 1.03 = 0.32g \\ \text{S} &= 4.12g \Rightarrow \frac{4.12}{16.66} = 0.25 \Rightarrow 0.25 \times 1.03 = 0.26g \end{aligned}$$

Total burning 45 min

Product: 1.17g WS: pWS 0.45g MB/B: passes sieve
0.22g sieved powderDeposit + Ynk 12.4g
Sieved 2.8g
Rod Remainder 1.6g
Left: crispy powder covered wallsSEM pWS: Curly bundles $\phi \sim 100$ nm embedded in amorph, very long ($> 5-10 \mu m$)TEM: Thin bundles of 5-20 tubes & some separate thick tubes of $\sim 4-5 \mu m$ ϕ . Most of tubes have 2 walls. Some are SWNTs (thick $\phi \sim 4-5 \mu m$). (The system selectively produces double-wall nanotubes in abundance!)

A. Morozky

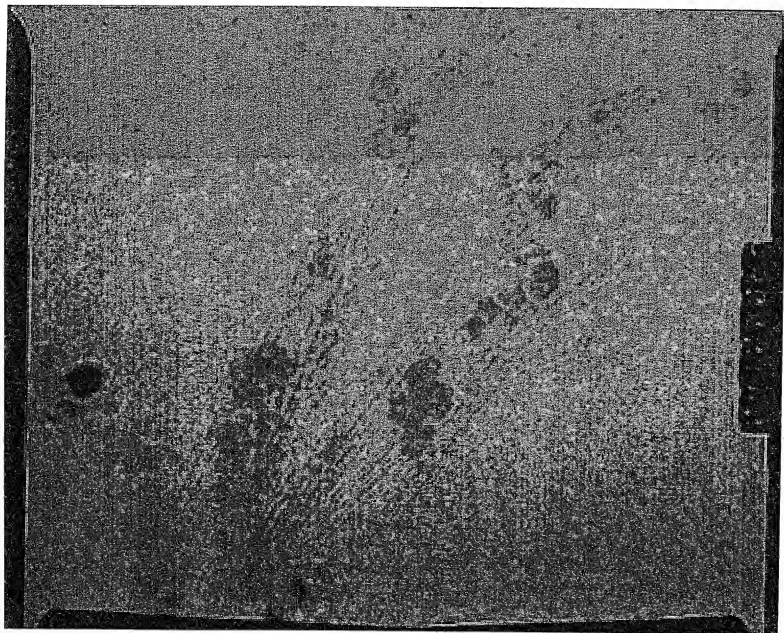
R. G. Kostin

11/1/98

EXHIBIT

3

TEM image of the product of Run CF39



EXHIBIT

4

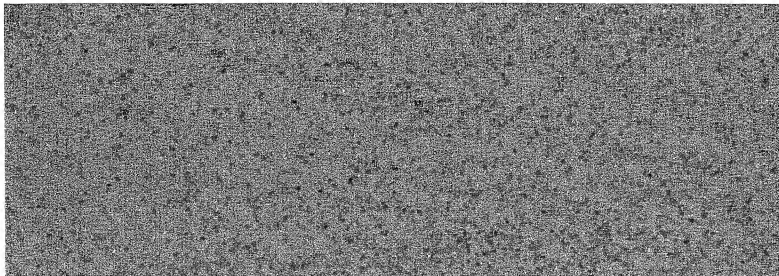
TEM image of the product of Run CF39



EXHIBIT

5

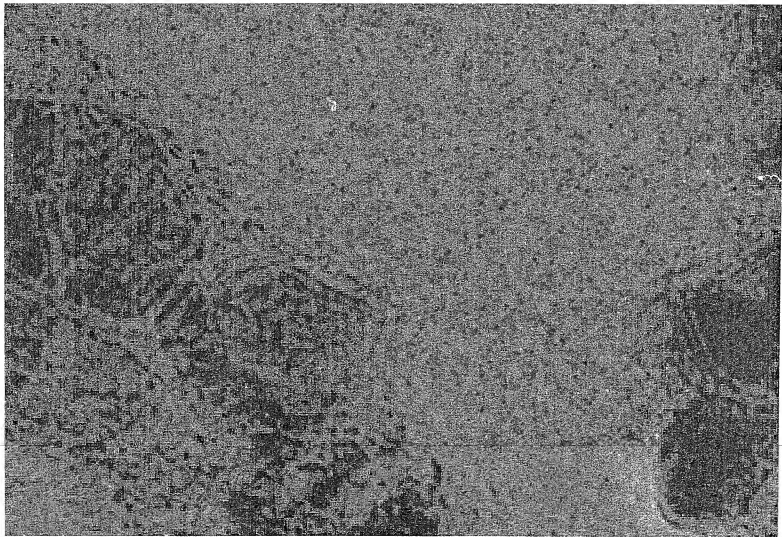
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EXHIBIT

6

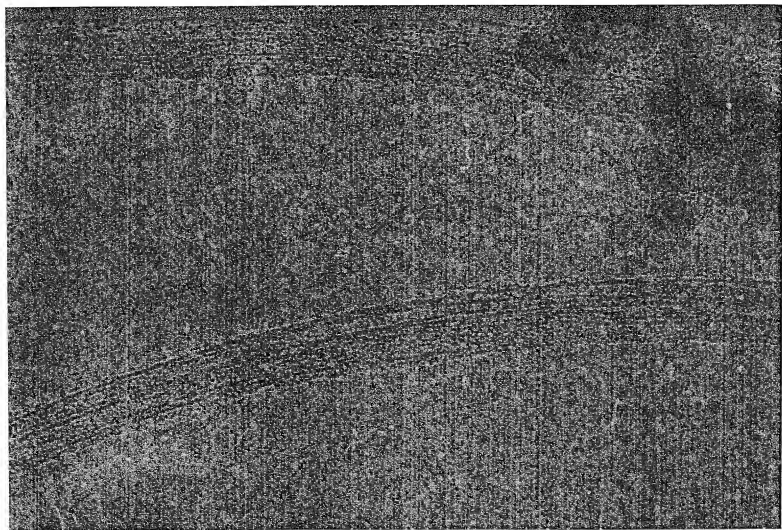
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EXHIBIT

7

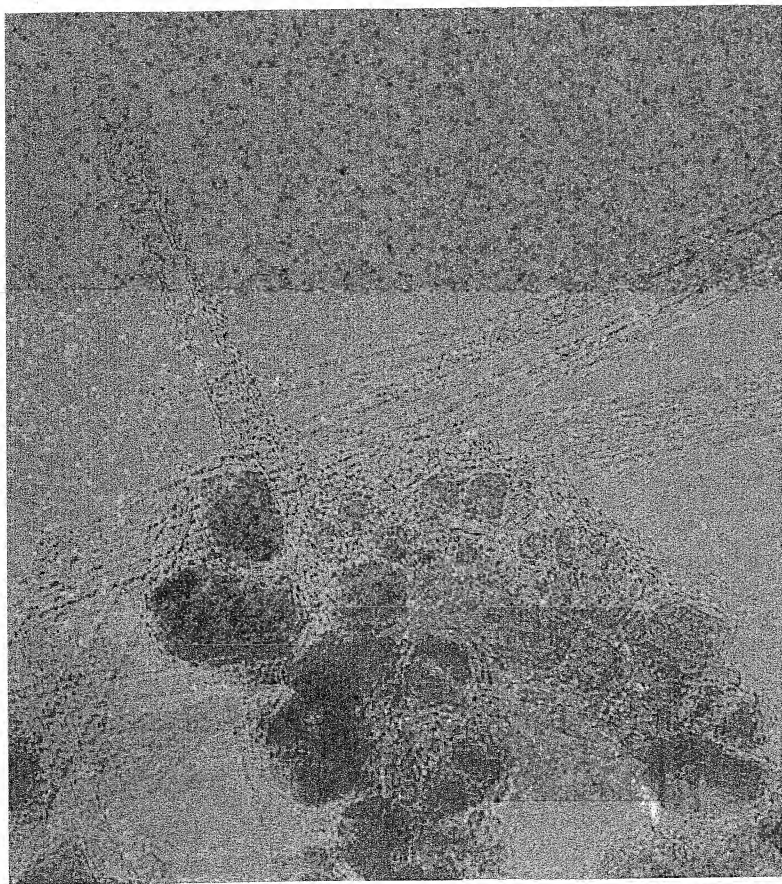
TEM image of the product of Run CF39



EXHIBIT

8

TEM image of the product of Run CF39



EXHIBIT

9

Message for Dr.A.P.Moravsky

Subject: Message for Dr.A.P.Moravsky

Date:

From: "Elena P. Krinichnaja" <elkrina@icp.ac.ru>

Organization: IPCP

To: "moravsky@Opus1.com" <moravsky@Opus1.COM>

Саша, здравствуй!

ТЭМ (кобальт, никель, железо и сульфид железа) двухслойные нанотрубки, диаметр 3.5–5.2 нм, расстояние между стенками 0.42+/-0.02 нм. Внутренние каналы нанотрубок частично заполнены, по всей видимости, аморфным углеродом. Помимо нанотрубок и каталитических частиц в образце присутствуют различные формы углерода, структура которых на данный момент времени не анализировалась.

Елка

П.С. Ты когда-нибудь ответишь мне?

EXHIBIT

10

E-mail message

for Dr. A.P.Moravsky from Dr.E.P.Krinichnaja

Translation into English

Hello, Alex!

TEM (cobalt, nickel, iron and iron sulfide catalyst) has shown: double-walled nanotubes of 3.5 – 5.2 nm diameters, with the distance between the walls 0.42 ± 0.02 nm.

Internal channels of nanotubes are partially filled, most likely, with amorphous carbon.

Besides the nanotubes and catalytic particles, there are present various forms of carbon with the structure that was not yet analyzed.

Elena.

P.S. Waiting for your reply.